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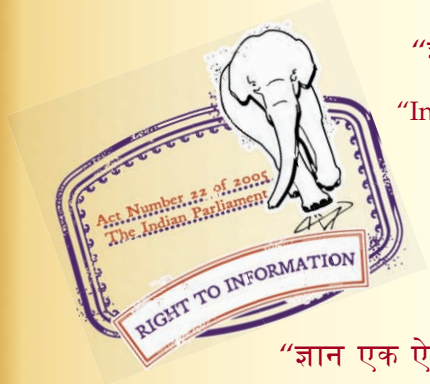

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Indian Standard

SPECIFICATION FOR DICHLORVOS EMULSIFIABLE CONCENTRATES

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SPECIFICATION FOR DICHLORVOS EMULSIFIABLE CONCENTRATES

(First Revision)

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AMENDMENT NO. 1 OCTOBER 1982

TO

IS : 5277-1978 SPECIFICATION FOR DICHLORVOS EMULSIFIABLE CONCENTRATES

(First Revision)

Alterations

(*Pages* 4 *and* 5, *clause* **2.3.1**) — Substitute the following for the existing clause:

'**2.3.1** *Dichlorvos Content* — When determined by the method prescribed in Appendix A, the observed dichlorvos content, percent (*m/m*), of any of the samples shall not differ from the nominal value by more than the percent tolerances indicated below:

Nominal Value, Percent	*Tolerance*
Up to 9	+ 10, − 5
Above 9 and below 50	± 5
50 and above	+ 5, − 3

percent of the nominal value'

(*Pages* 9 *and* 10, *clause* **A-2.5**) — Substitute the following for th existing matter:

'**A-2.5 Estimation of Dichlorvos** — Cover the perforated disc of chromatographic column (*see* **A-2.2.4**) with glass wool pad and fill the column to a height of 50 mm with chloroform. Weigh 3 g cellulose chromatographic grade (*see* **A-2.3.3**) in a 100 ml beaker and add to it 0·2 ml distilled water. Stir with glass rod for uniform mixing. Add 20 ml chloroform to the beaker and transfer quantitatively to the chromatographic column with additional quantity of chloroform. Gently tap the column for uniform compacting.

A-2.5.1 Weigh 0·25 g of sample and dissolve in chloroform. Transfer quantitatively to the chromatographic column when the level of solvent is just above the solid phase. Collect the eluted solvent in a beaker placed below the constricted end. Elute with additional 100 ml chloroform, adding it in 10 ml portion. Evaporate the eluted solvent on a

water bath to 5 ml and transfer it to a 10 ml volumetric flask. Wash the beaker with 2 ml portions of chloroform and add washings to the volumetric flask. Make up the volume to 10 ml with chloroform. Mix thoroughly and fill the calibrated liquid absorption cell with the sample solution. Using the same instrument settings that were used for the calibration obtain replicate scan of the sample solution over the 9·9 to 10·6 micron region. Calculate the absorbance of the sample solution for the two reference minima as described in **A-2.4.1.4.**

A wide range spectra of sample over a wider wavelength may be compared with the spectrum of pure dichlorvos and it should be ensured that no undesirable peaks are present in the region of 10 microns and 11 microns.'

Addenda

(*Page* 5, *clause* **2.3.1.1**) — Add the following new clause after **2.3.1.1:**

'**2.3.1.2** The average content of all samples taken shall not be lower than the nominal content.'

(*Page* 9, *clause* **A-2.2.3**) — Add the following new clause after **A-2.2.3:**

'**A-2.2.4** *Chromatographic Column* — made of glass, length 250 mm and internal diameter 20 mm, fitted with a perforated disc and constricted to 5 mm diameter at the lower end.'

(*Page* 9, *clause* **A-2.3.2**) — Add the following new clause after **A-2.3.2:**

'**A-2.3.3** *Cellulose* — Chromatographic grade.'

(AFCDC 6)

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1983. When the material is offered for inspection after 90 days of its manufacture, sampling shall be done as prescribed in IS 10627 : 1983. However, the criteria for conformity of the material shall be the limits of tolerences given under clause 2.3.1 of the standard.'

(*Page* 6, *clause* 5.2) — Substitute 'IS 1070 : 1992*' *for* 'IS : 1070 - 1977*'.

(*Page* 6, *foot-note with* '*' *mark*) — Substitute '*Reagent grade water (third revision)*' for the existing title.

(FAD 1)

Reprography Unit, BIS, New Delhi, India

AMENDMENT NO. 2 JUNE 1990
TO
IS 5277:1978 SPECIFICATION FOR DICHLORVOS EMULSIFIABLE CONCENTRATES

(First Revision)

(Cover page, pages 1 and 3, title) - Substitute 'Dichlorvos EC' for 'Dichlorvos emulsifiable concentrates' from the title and wherever it occurs in the text of the standard.

(Page 3, clause 0.2) - Substitute the follow' ; for the existing clause:

'Dichlorvos EC are used as insecticides and acaricides in agriculture. The material is also used for out-door fly control by recognised agencies like the Municipalities, Railways. It is not used for domestic fly control.'

(FAD 1)

Reprography Unit, BIS, New Delhi, India

AMENDMENT NO. 3 DECEMBER 1995
TO
IS 5277 : 1978 SPECIFICATION FOR DICHLORVOS EC

(First Revision)

(*Page* 3, *clause* 0.4) — Substitute 'IS : 6940 - 1982*' *for* 'IS : 6940 - 1973*'.

(*Page* 3, *foot-note marked* '*') — Insert '(*first revision*)' at the end of text.

(*Page* 4, *clause* 2.2.2) — Substitute 'IS : 6940 - 1982†' *for* 'IS : 6940 - 1973†'.

(*Page* 4, *foot-note marked* '†') — Insert '(*first revision*)' at the end of text.

(*Page* 4, *clause* 2.2.3) — Substitute 'IS 1448 (P:20) : 1982‡' *for* 'IS : 1448 (P : 20) - 1960‡'.

(*Page* 4, *foot-note marked* '‡') — Insert '(*first revision*)' at the end of text.

(*Page* 4, *clause* 2.2.4) — Substitute 'IS : 6940 - 1982†' *for* 'IS : 6940 - 1973†'.

(*Page* 5, *clause* 2.3.2) — Substitute 'IS : 6940 - 1982*' *for* 'IS : 6940 - 1973*'.

(*Page* 5, *foot-note marked* '*') — Insert '(*first revision*)' at the end of text.

(*Page* 5, *clause* 3.1) — Substitute 'IS : 8190 (Part 2) - 1988†' *for* 'IS : 8190(Part II) - 1976†'.

(*Page* 5, *foot-note marked* '†') — Insert '(*second revision*)' at the end of text.

(*Page* 6, *clause* 4.1) — Substitute the following for the existing:

'When bulk manufactured material is offered for inspection, representative samples of the material shall be drawn and tested as prescribed in IS 10627 : 1983 'Methods of sampling of pesticides and their formulations' within 90 days of its manufacture. The criteria for conformity shall be as given in IS 10627 :

AMENDMENT NO. 4 MAY 2002
TO
IS 5277 : 1978 SPECIFICATION FOR DICHLORVOS EMULSIFIABLE CONCENTRATES

(First Revision)

(*Page 8, clause* A-2.2.2, *line* 1) — Substitute 'sodium chloride or potassium bromide' *for* 'sodium chloride'

(FAD 1)

Reprography Unit, BIS, New Delhi, India

AMENDMENT NO. 5 OCTOBER 2003
TO
IS 5277 : 1978 SPECIFICATION FOR DICHLORVOS EMULSIFIABLE CONCENTRATES

(First Revision)

[*Page* 5, *clause* 2.3.2 (*see also Amendment No.* 3) — Substitute '13.5 of IS 6940 : 1982' *for* '11.5 of IS 6940 : 1973*'.

(FAD 1)

Reprography Unit, BIS, New Delhi, India

Indian Standard

SPECIFICATION FOR DICHLORVOS EMULSIFIABLE CONCENTRATES

(First Revision)

0. FOREWORD

0.1 This Indian Standard (First Revision) was adopted by the Indian Standards Institution on 15 February 1978, after the draft finalized by the Pest Control Sectional Committee had been approved by the Agricultural and Food Products Division Council and the Chemical Division Council.

0.2 Dichlorvos emulsifiable concentrates are largely used in the control of insects, pests and mites of agricultural importance.

0.3 Dichlorvos emulsifiable concentrate is generally manufactured to contain 76 percent (*m/m*) of dichlorvos.

0.4 This standard was first published in 1969. Subsequently four amendments were issued. This revision incorporates the revised requirements pertaining to description, emulsion stability, packing, cautionary notice and sampling. Opportunity has been taken to give the reference to IS : 6940-1973* to bring a uniform testing procedure to the various requirements in vouge.

0.5 In the preparation of this standard, due consideration has been given to the provisions of the Insecticides Act, 1968 and the Rules framed thereunder. However, this standard is subject to the restrictions imposed under these, wherever applicable.

0.6 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960†. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

*Methods of tests for pesticides and their formulation.

†Rules for rounding off numerical values (*revised*).

1. SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for dichlorvos emulsifiable concentrates.

2. REQUIREMENTS

2.1 Constituents — The material shall consist of dichlorvos, technical dissolved in suitable solvent(s), together with emulsifying agent(s), and with or without stabilizer(s) and suitable dye(s).

2.1.1 Dichlorvos, technical, employed in the manufacture of this material shall conform to IS : 4929-1978*.

2.2 Physical — The material shall comply with the physical requirements given in **2.2.1** to **2.2.4**.

2.2.1 *Description* — The material shall be in the form of a homogeneous stable liquid free from sediment. Suspended impurities shall be negligible.

2.2.2 *Cold Test* — No turbidity or separation of solid and/or oily matter shall occur when the material is subjected to the cold test at 10°C as prescribed in **13.1** of IS : 6940-1973† or any other lower temperature as agreed to between the purchaser and the vendor.

2.2.3 *Flash Point (Abel)* — When determined by the method prescribed in IS : 1448 [P : 20]-1960‡, the flash point of the material shall be above 24·5°C.

2.2.4 *Emulsion Stability* — Any separation, including creaming at the top and sedimentation at the bottom, of 100 ml of emulsion prepared in standard hard water with 5 ml for public health use and 2 ml for agricultural use, shall not exceed 2 ml when tested by one of the methods prescribed in **13.3** of IS : 6940-1973†.

2.3 Chemical — The material shall comply with the chemical requirements given in **2.3.1** and **2.3.2**.

2.3.1 *Dichlorvos Content* — When determined by the method prescribed in Appendix A, the observed dichlorvos content, percent (*m/m*), of any of

*Specification for dichlorvos, technical (*first revision*).
†Methods of tests for pesticides and their formulation.
‡Methods of test for petroleum and its products, P : 20 Flash point by abels apparatus.

the samples shall not differ from the nominal value by more than the percent tolerances indicated below:

Nominal Value	*Tolerance on the Nominal Value, Percentage*
Up to 9	$\begin{matrix}+10\\-5\end{matrix}$
10 and below 50	$\pm$ 5
50 and above	$\begin{matrix}+5\\-3\end{matrix}$

2.3.1.1 The actual value of the dichlorvos content in the formulation shall be calculated to the second decimal place and then rounding off to the first decimal place before applying the tolerances as given in **2.3.1.**

2.3.2 *Acidity* — When tested by the method prescribed in **11.3** of IS : 6940-1973*, the acidity (as H_2SO_4) of the material shall be not more than 1·0 percent by mass.

2.3.2.1 For determination of acidity, the end point can also be determined by a potentiometric titration to *p*H 5·0.

3. PACKING AND MARKING

3.1 Packing — The material shall be packed according to IS : 8190 (Part II)-1976†.

3.2 Marking — The containers shall bear legibly and indelibly the following information and any other additional information as is necessary under the Insecticides Act and Rules:

a) Name of the material;

b) Name of the manufacturer;

c) Date of manufacture;

d) Batch Number;

e) Net volume of contents;

f) Nominal dichlorvos content, percent (*m/m*); and

g) The cautionary notice worded as in the Insecticides Act and Rules.

---

*Methods of tests for pesticides and their formulation.

†Requirements for packing of pesticides: Part II Liquid pesticides.

3.2.1 The containers may also be marked with the ISI Certification Mark.

NOTE — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act and the Rules and Regulations made thereunder. The ISI Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

4. SAMPLING

4.1 Representative samples of the material shall be drawn as prescribed in 'Indian Standard methods for sampling of pesticides and their formulations' (*under preparation*).

NOTE — Till such time the standard under preparation is published the matter shall be as agreed to between the concerned parties.

5. TESTS

5.1 Tests shall be carried out by the appropriate methods referred to in **2.2.1** to **2.2.4**, **2.3.1** and **2.3.2**.

5.2 Quality of Reagents — Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1977*) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

APPENDIX A

(*Clause* 2.3.1)

DETERMINATION OF ACTIVE INGREDIENT CONTENT

A-0. GENERAL

A-0.1 For the determination of percent content of dichlorvos, technical, in dichlorvos emulsifiable concentrates, two methods, namely, the mercaptometric method and infra-red spectrophotometric method, have been prescribed. Either of these methods may be used. However, in the event of dispute, the infra-red spectrophotometric method shall be used as the reference method.

*Specification for water, for general laboratory use (*second revision*).

A-1. MERCAPTOMETRIC METHOD

A-1.1 Principle of Method — Acid degradation products and aldehydes are removed by an aqueous solution of sodium hydrogen sulphite and the dichlorvos titrated with mercaptan in a solution of benzene. Mercaptan reacts with dichlorvos by splitting off a methyl group.

A-1.2 Reagents

A-1.2.1 *Isopropyl Alcohol*

A-1.2.2 *Sodium Hydrogen Sulphite*

A-1.2.3 *Benzene*

A-1.2.4 *Saturated Sodium Bicarbonate Solution*

A-1.2.5 *Anhydrous Sodium Sulphate*

A-1.2.6 *Normal Dodecylmercaptan Solution* — Dissolve 2·5 ml of technical dodecylmercaptan in 100 ml of isopropyl alcohol. The solution is stable for few days, it degrades within a week by about 10 percent.

A-1.2.7 *Tetramethyl Ammonium Hydroxide Solution* — 1·5 percent (*m/v*). Dilute 15 ml of 10 percent (*m/v*) aqueous solution with isopropyl alcohol to 100 ml. A fresh solution has to be prepared every day.

A-1.2.8 *Glacial Acetic Acid*

A-1.2.9 *Iodine Solution* — 0·1 N.

A-1.3 Procedure

A-1.3.1 Weigh accurately about 2·5 g of the concentrate and dissolve in 30 ml of distilled water and 15 ml of isopropyl alcohol. To this solution add 3·0 g of sodium hydrogen sulphite. Shake well until completely dissolved and allow to stand for 15 minutes.

A-1.3.2 Transfer the solution to a separating funnel, add 20 ml of distilled water and 75 ml of benzene, shake well for 1 minute and allow to separate. Extract the aqueous phase twice more with successive lots of 50 ml of benzene. To remove any sulphur dioxide, which might be present, shake the benzene extracts with 20 ml of a saturated solution of sodium bicarbonate for not more than 30 seconds. For this purpose, combine the first two extracts, shake with sodium bicarbonate solution and allow to separate. The third benzene extract is washed with the same sodium bicarbonate solution. Dilute the combined benzene extracts to 200 ml, mix well. Discard certain quantity, then add to the rest of the solution 25 g of anhydrous sodium sulphate. Filter the solution through filter paper, reject the first 20 ml of filtrate.

A-1.3.3 To 20 ml of the filtered solution in a 100-ml ground glass stoppered Erlenmeyer flask, add 25 ml of dodecylmercaptan solution. Let the pipette drain for 30 seconds. Replace the air in the flask by nitrogen and 10 ml of the tetramethyl ammonium hydroxide solution and close the flask immediately. Allow to stand for 15 minutes at about 25°C. Acidify with 5 ml of glacial acetic acid and titrate with 0·1 N iodine solution to the first permanent yellowish colour.

A-1.3.4 Carry out a blank titration using 20 ml of benzene, 25 ml of dodecylmercaptan solution and 10 ml of tetramethylammonium hydroxide solution. Allow to stand for 15 minutes at about 25°C. Acidify with 5 ml of glacial acetic acid and titrate with 0·1 N iodine solution to the first permanent yellowish colour.

A-1.4 Calculation

$$\text{Dichlorvos content, percent by mass} = \frac{22{\cdot}10\,(B - A)\,F}{m}$$

where

B = ml of iodine solution required in the blank titration,

A = ml of iodine solution required in the first titration (*see* Note),

F = factor of iodine solution,

m = mass, in g, of the concentrate taken for the test.

NOTE — One millilitre of 0·1 N iodine solution is equivalent to 22·10 mg of dichlorvos.

A-2. INFRA-RED SPECTROPHOTOMETRIC METHOD

A-2.1 Principle of Method — The method consists in dissolving the material in chloroform, and the infra-red absorbance measured at about 10·2 μm employing a reference-point technique. This net absorbance is used to obtain the concentration of dichlorvos from previously prepared calibration curve relating to net absorbance concentration of dichlorvos.

A-2.2 Apparatus

A-2.2.1 *Infra-red Spectrophotometer* — capable of recording in the region of 2 to 15 μm, with the slit width, gain and response time and scanning speed adjustable to produce a satisfactory signal-to-noise ratio and adequate resolution under the conditions of the test (in general, the minimum slit width giving a signal-to-noise ratio of about 100 to 1 is chosen).

A-2.2.2 *Absorption Cells* — sealed absorption cells with sodium chloride windows, having a path length of about 0·2 mm.

A-2.2.3 *Hypodermic Syringe* — of 1·0 ml capacity with 1·25 mm (stubbs) slip-on-type needle.

A-2.3 Reagents

A-2.3.1 *Standard Dichlorvos* — recrystallized, of known dichlorvos content.

A-2.3.2 *Chloroform*

A-2.4 Procedure

A-2.4.1 *Preparation of Calibration Graph* — Prepare the calibration graph for samples in chloroform according to A-2.4.1.1 to A-2.4.1.4.

A-2.4.1.1 Weigh accurately into each of the five 10-ml volumetric flasks 25, 75, 100, 150 and 200 mg of the standard dichlorvos (*see* A-2.3.1), dissolve in chloroform and dilute to the mark (the concentrations of these solutions will be 2·5, 7·5, 10, 15 and 20 g per 1 litre).

A-2.4.1.2 Fill the absorption cell with chloroform by means of the hypodermic syringe. Adjust the spectrophotometer to the optimum instrument settings with respect to gain, slit width, response, chart speed and wave-length scanning speed. Make a scan with chloroform in the cell over the wave-length region of 9·9 to 10·6 µm.

A-2.4.1.3 Without changing the instrument settings, fill the cell, in turn, with each of the calibration solutions starting with the most dilute. Scan each of these solutions over the 9·9 to 10·6 µm wave-length region.

A-2.4.1.4 For each of the scans obtained draw perpendiculars to the zero radiation line through the absorption peak of the calibration solution at about 10·2 µm and the reference minima at about 10·0 µm and 10·4 µm measure the radiant power P_o and P as shown in Fig. 1. The distances may be measured in any convenient units, provided the same units are used throughout the determination. Calculate the absorbance as the logarithm of the ratio of the incident power (P_o) to the transmitted radiant power (P). Repeat the calculation of the absorbances of the calibration solutions using the reference minimum at about 10·4 µm. Subtract the absorbance of the cell plus calibration solutions. Make a plot of the net absorbances as ordinate against the corresponding concentrations of dichlorvos in g/litre as abscissa for each of the reference points used, that is the absorption minima at about 10·0 µm and 10·4 µm respectively.

A-2.5 **Estimation of Dichlorvos** — Weigh accurately into a 50-ml volumetric flask an amount of sample sufficient to give 1 percent (*m/v*) solution of dichlorvos and dilute to the mark with chloroform. Mix thoroughly and fill the calibrated liquid absorption cell with the sample solution. Using the same instrument settings that were used for the calibration, obtain a scan of the sample solution over the 9·9 to

10·6 micron region. Calculate the absorbances of the sample solution for the two reference minima as described in A-2.4.1.1.

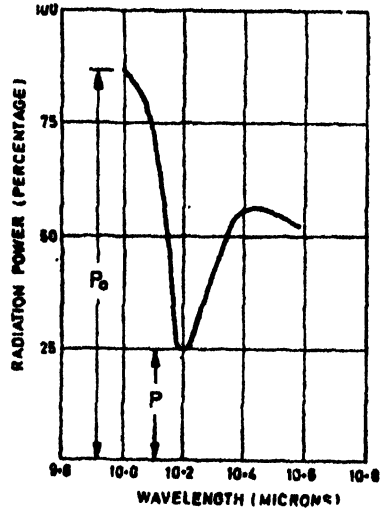

FIG. 1 DICHLORVOS INFRA-RED ABSORPTION SPECTRUM-REFERENCE-POINT TECHNIQUE

A-2.6 Calculation — From the computed absorbances (*see* A-2.5) read the concentrations of dichlorvos, technical, from calibration graph (*see* A-2.4.1.1).

$$\text{Active ingredient content, percent by mass} = \frac{A \times V}{M \times 10}$$

where

A = the mean value, in g, per 1 litre determined from the use of two reference minima;

V = volume, in ml, of the sample solution; and

M = mass, in g, of the sample.

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